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METHODS OF
CHEMICAL ANALYSIS OF FERROSILICON

PART IV DETERMINATION OF PHOSPHORUS

(First Revision)

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METHODS OF CHEMICAL ANALYSIS OF FERROSILICON

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METHODS OF CHEMICAL ANALYSIS OF FERROSILICON

PART IV DETERMINATION OF PHOSPHORUS

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 29 January 1982, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 IS : 1559-1961* which covers chemical analysis of different ferro-alloys, namely, ferrosilicon, ferrochromium, ferromanganese and speigeleisen, silico-manganese, ferromolybdenum, ferrophosphorus, ferrotitanium, ferrovanadium and ferrotungsten, was first published in 1961. While reviewing this standard, the Sectional Committee felt that the standard should be revised to cover ferrosilicon, and methods of analysis of other ferro-alloys should be covered in separate standards. During preparation of the revision of IS : 1559-1961* accordingly, it was felt convenient to cover methods of chemical analysis of ferrosilicon in different parts of the standard which on publication would supersede Section I ferrosilicon of IS : 1559-1961*. The committee also decided to rewrite the methods taking into consideration the latest composition specified in IS : 1110-1981†. Though calcium and manganese are not specified in IS : 1110-1981†, the determination of these elements are covered in this standard because these are generally required from the view point of determining interference of analysis in methods for determining other elements in ferrosilicon due to presence of these elements. Moreover the determination of these elements may be required for comprehensive analysis of ferrosilicon. This part covers the determination of phosphorus. The other parts cover as follows:

Part I	Determination of silicon
Part II	Determination of carbon

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*Methods of chemical analysis of ferro-alloys.

†Specification for ferrosilicon (*third revision*).

Part III	Determination of sulphur
Part V	Determination of aluminium
Part VI	Determination of calcium
Part VII	Determination of manganese

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part IV) covers the determination of phosphorus (0·050 to 0·15 percent) in ferrosilicon.

2. QUALITY OF REAGENTS

2.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

3. SAMPLING

3.1 It shall be drawn and prepared in accordance with IS : 1472-1977‡.

4. DETERMINATION OF PHOSPHORUS BY THE VOLUMETRIC (ALKALIMETRIC) METHOD

4.1 Outline of the Method — The sample is dissolved in nitric and hydrofluoric acids in a platinum dish. The solution is evaporated to low bulk and the salts are dissolved in dilute nitric acid. The solution is filtered. From the filtrate, phosphorus is precipitated as ammonium phosphomolybdate in presence of boric acid estimated by the alkalimetric method.

4.2 Reagents

4.2.1 *Concentrated Nitric Acid* — rd = 1·42 (conforming to IS : 264-1976§).

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*Rules for rounding off numerical values (*revised*).

†Specification for water for general laboratory use (*second revision*).

‡Method of sampling ferro-alloys, for determination of chemical composition (*first revision*).

§Specification for nitric acid (*second revision*).

4.2.2 *Hydroflouric Acid* — 40 percent and rd = 1·13.

4.2.3 *Dilute Potassium Permanganate Solution* — 2 percent (*m/v*).

4.2.4 *Sodium Nitrite Solution* — 5 percent (*m/v*).

4.2.5 *Boric Acid* — solid.

4.2.6 *Ammonium Hydroxide* — rd = 0·90 and 50 percent.

4.2.7 *Ammonium Molybdate Solution*

a) Weigh 50 g of ammonium molybdate in a beaker, add 200 ml of ammonium hydroxide, dissolve and keep it separately; and

b) Mix 400 ml of concentrated nitric acid and 400 ml of water in a 2-litre conical flask, cool and keep it separately.

Add slowly solution (*a*) to solution (*b*) with continuous shaking and cooling. To this solution, add about 0·1 g of ammonium phosphate, and allow for settlement. Filter and use for precipitation of phosphorus.

4.2.8 *Potassium Nitrate Wash Solution* — 1 percent (*m/v*). Solution should be prepared with carbon-dioxide free water.

4.2.9 *Nitric Acid Wash Solution* — 1 percent (*v/v*).

4.2.10 *Sodium Hydroxide Solution (Approximately 0·075 N)* — Dissolve 3 g of sodium hydroxide in 1 litre of water.

4.2.11 *Standard Nitric Acid (0·075 N)* — Add 4·7 ml of concentrated nitric acid (rd = 1·42) to 1 litre of water and standardize against standard sodium carbonate solution using methyl orange as indicator.

4.2.12 *Standard Sodium Carbonate Solution* (0·075 *N*) — Weigh exactly 0·397 5 g of sodium carbonate, preheated to 250° to 270°C for half-an-hour and cool in 100-ml volumetric flask. Dissolve in distilled water and make up to the mark. Standardize against standard nitric acid using methyl orange.

4.2.13 *Methyl Orange* — Dissolve 0·05 g in 100 ml of distilled water.

4.2.14 *Phenolphthalein* — Dissolve 0·5 g of the reagent in 50 ml of alcohol and dilute with 50 ml of distilled water with constant stirring.

5. PROCEDURE

5.1 Dissolve 2 g of dried sample in a platinum dish, using 20 ml of concentrated nitric acid and add hydrofluoric acid dropwise until the vigorous reaction has subsided. Heat to complete dissolution and evaporate to low bulk. Add 30 ml of concentrated nitric acid and a few ml of dilute potassium permanganate solution till permanganate colour persists.

Boil and destroy excess potassium permanganate solution with sodium nitrite solution. Dilute to about 50 ml, filter and wash. Collect the filtrate and washings in a 500-ml conical flask. Add about 2 g of boric acid to the solution in the flask and heat to dissolve. Add ammonium hydroxide (50 percent) till the precipitate formed difficultly dissolves. Dissolve the precipitate in concentrated nitric acid added dropwise finally providing 5 ml in excess. Heat the solution to boiling on a hot-plate and then keep it aside to attain a temperature of 80°C. Add now 30 to 35 ml of freshly filtered ammonium molybdate solution and shake vigorously for about 15 minutes. Keep the solution for settlement for about half-an-hour.

5.2 Filter the precipitate of ammonium phospho-molybdate on a paper pulp pad transferring all the precipitate from the flask into the pad by washing with cold neutral potassium nitrate wash solution. Wash the precipitate on the pad three times with cold nitric acid wash solution and then with cold neutral potassium nitrate wash solution again until the washings are no longer acid to phenolphthalein (*see* Note). Transfer the precipitate to the original conical flask where the phosphorus was first precipitated. Add a known excess of sodium hydroxide solution. Titrate the excess of sodium hydroxide solution with standard nitric acid by using phenolphthalein as indicator. Take a blank of sodium hydroxide solution used in the experiment and from the difference calculate the percentage of phosphorus as given in **6.**

NOTE — 10 ml of final washings collected in a test tube should give pink colour with phenolphthalein by a drop of sodium hydroxide solution of approximately 0·075 N. Normally the precipitate is washable free from acid by giving about 5 to 6 washings.

6. CALCULATION

$$\text{Phosphorus, percent} = \frac{(V_1 - V_2) \times N \times 0{\cdot}001\,35 \times 100}{M}$$

where

V_1 = volume in ml of standard nitric acid required by the blank,

V_2 = volume in ml of standard nitric acid required in the experiment,

N = strength of standard nitric acid used, and

M = mass in g of the sample taken.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	$1\ \text{N} = 1\ \text{kg.m/s}^2$
Energy	joule	J	$1\ \text{J} = 1\ \text{N.m}$
Power	watt	W	$1\ \text{W} = 1\ \text{J/s}$
Flux	weber	Wb	$1\ \text{Wb} = 1\ \text{V.s}$
Flux density	tesla	T	$1\ \text{T} = 1\ \text{Wb/m}^2$
Frequency	hertz	Hz	$1\ \text{Hz} = 1\ \text{c/s}\ (\text{s}^{-1})$
Electric conductance	siemens	S	$1\ \text{S} = 1\ \text{A/V}$
Electromotive force	volt	V	$1\ \text{V} = 1\ \text{W/A}$
Pressure, stress	pascal	Pa	$1\ \text{Pa} = 1\ \text{N/m}^2$

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